

FURACA 3.10

AIM: Preparation of FURACA from TACA by New Route

STAGE-I: PREPARATION OF ~~EA~~ TFA (STD).

Raw Materials:

NaSH : 37.5 g

DMN : 365.0 g

2-Furyl Chloride : 27.5 g

EtoAc : (250+100) ml

NaHCO<sub>3</sub> : 20.0 g

DMN : 175.0 ml

for HCl 1:1 HCl : (48+49) ml

PROCEDURE:

1. Charge DMN, cool to  $25-20^{\circ}\text{C}$ .
2. Charge NaSH and stir 5' to get clear soln.
3. Add Furyl chloride in 40-45' at 20-25°C and then stir 10' at same temp.
4. Then charge EtoAc (250ml) and adjust the pH to 0.9-1.0 with 1:1 HCl at 20-25°C in 15'.
5. Separate the layers. Eliminate the aq. phase.
6. To the org. phase charge DMN (175) and adjust the pH to 7.0-7.1 with NaHCO<sub>3</sub> at 20-22°C in 15'.
7. Then stir for 30' at 20-22°C and separate the layers.
8. To the rich aq. layer charge 100 ml EtoAc and again adjust the pH to 0.9-1.0 with 1:1 HCl at 20-22°C in 15'.
9. Stir 15' at 20-22°C and separate the layers.
10. This Org. layer (O<sub>1</sub>) is taken for next stage.

Vol. of O<sub>1</sub> (TFA) : ~~130.0~~ ml

HCl	TFA	Imp.
O <sub>1</sub>	97.34	(1.0+1.0)

O <sub>1</sub>	97.48	2.1
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STAGE-II: FURACA PREPARATION:

Modi : (A) STD. + Etone slurry given separately and spray

(B) pH is adjusted to 3.0 only and the wet cake was washed with Etone slurry and spray.

Raw Materials

7AcA : 50.0 g

Etone : 200.0 g

GAA : 30.0 ml

$\text{BF}_3$  gas : 68.5 g

TFA sol<sup>n</sup> : 130.0 ml

EDTA : 0.3 g

(A)

(B)

Rxn. mass

DMW	75.0 ml	75.0 ml
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EDTA	0.15 g	0.15 g
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SHS	0.5 g	0.5 g
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18-20% NH <sub>3</sub> sol	48.0 ml 25ml	42.0 ml 3.0 ml
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DMW	(25+75+25) ml	(25+75+25) ml
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Eton	(75+25) ml	(75+25) ml
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PROCEDURE:

1. Charge Etone, GAA at RT and cool to 0°C.
2. Purge  $\text{BF}_3$  gas at  $\leq 10^\circ\text{C}$ .
3. Add EDTA and stir 35' at  $15^\circ\text{C}$ .
4. Then charge 7AcA + stir for 5' and then charge TFA sol<sup>n</sup>.
5. Stir of the Rxn. mixture at  $30^\circ\text{C}$  for  $2\frac{1}{2}$  hr. & monitor the Rxn. mass.  
Divide the mass into two equal parts.

Chare

(A) ① Boil the one part into the precooled DMW (15°C) and add EDTA + SHS.

② Adjust the pH to 3.5 by 18-20% NH<sub>3</sub> sol. in 40-45' at 25-30°C.

③ Stir for 30' at 25°C and then filter it.

④ Wash the product with DMW spray, slurry & spray.

⑤ Take the wet cake in a RBF and add 75ml and stir for 15' at 25°C and then filter.

⑥ Then give 25ml Etone spray.

⑦ Dry the product at 30-35°C for 2hr-3hr.

⑧ Quantify the ~~Etone~~ washing Etone layer.

(B) ① Transfer the Remain (another part) into the 15°C pH DMW and add EDTA + SHS.

② Adjust the pH to 3.0 by 18-20% NH<sub>3</sub> sol. in 40-45' at 25-30°C.

③ Stir for 20' at 25°C and then filter it.

④ Wash the product with DMW spray, slurry & spray.

⑤ Take the wet cake into the RBF and add 75ml Etone and stir 15' at 25°C.

⑥ Filter it & wash with 25ml Etone spray.

⑦ Dry it at 25°C for 2-3hr.

⑧ Quantify the washing Etone layer.

R/M Results

Duration TAcA FURAcA TFA Dmp

1hr. 4.96 81.95 11.70 0.35

2 hr. 0.74 89.86 7.81 0.33

2 1/2 hr. 0.20 91.26 7.10

Terminated in 2 1/2 hr.

OBS: (1) TFA having 2% Impurity, even though FURACe Ben. gone smoothly.

(2) Colour of the Ben. man is normal.

(3) While ~~pH~~  $\text{NH}_3$  addition, the colour of (B) slurry is somewhat different, it is in greenish colour.

(4) In both case Rate of filtration is normal & material ~~normal~~ nature also same.

	Crude Net wt	Etope slurry + spray	Washed wet wt	Dry wt.
(A)	61.2 g	54.3 g	28.4 g	
(B)	60.3 g	56.4 g	29.5 g	

Etope slurry + spray washed,  $\text{exg}$  Etope layer wts % of FURACe

(A) Wt. of Etope only : 54.5 g / 60.0 ml

(B) Wt. of Etope only : 35.4 g / 40.0 ml

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HPLC Report:

	FURACe	TACA	TFA	Imp.	CV
(A) Crude	97.37	0.35	1.7	0.31	
(B) Crude Etope washed	90.91	0.19	2.15	0.10	
(B) Etope					
Etope washed					

Quantitative Analysis:

	FURACe	mlc	% of FURACe in Eto.
(A) Purified	91.93	2.1	0.0033%
(B) "	81.84	2.01	0.0022%